DB Name	Query	Hit Count	Set Name
USPT,PGPB,JPAB,EPAB,DWPI,TDBD	l4 and l12	2	L14
USPT,PGPB,JPAB,EPAB,DWPI,TDBD	l4 and l11	2	L13
USPT,PGPB,JPAB,EPAB,DWPI,TDBD	l10 and l3	5	L12
USPT,PGPB,JPAB,EPAB,DWPI,TDBD	l10 and l2	26	L11
USPT,PGPB,JPAB,EPAB,DWPI,TDBD	sealant or adhesive	731119	L10
USPT,PGPB,JPAB,EPAB,DWPI,TDBD	l1 and l3 and l4	0	L9
USPT,PGPB,JPAB,EPAB,DWPI,TDBD	l1 and l2 and l4	0	L8
USPT,PGPB,JPAB,EPAB,DWPI,TDBD	l1 and (l2 or l3) and l4	0	L7
USPT,PGPB,JPAB,EPAB,DWPI,TDBD	l1 and (l2 or l3)	3	L6
USPT,PGPB,JPAB,EPAB,DWPI,TDBD	tackifier	17519	L5
USPT,PGPB,JPAB,EPAB,DWPI,TDBD	tackifier	17519	L4
USPT,PGPB,JPAB,EPAB,DWPI,TDBD	hydrogenated styrene isoprene copolymer	46	L3
USPT,PGPB,JPAB,EPAB,DWPI,TDBD	hydrogenated styrene butadiene copolymer	162	L2
USPT,PGPB,JPAB,EPAB,DWPI,TDBD	pressure sensitive (sealant or adhesive)	40832	L1

5. The process of claim 1 wherein said disulfide of formula I is selected from the group consisting of 2,2'-bis(trimethoxysilylethyl) disulfide; 3,3'-bis(trimethoxysilylpropyl) disulfide; 3,3'-bis(triethoxysilylpropyl) disulfide; 2,2'-bis(tripropoxysilylethyl) disulfide; 2,2'-bis(triethoxysilylpropyl) disulfide; 3,3'-bis(triethoxysilylpropyl) disulfide; 2,2'-bis(trisec.butoxysilylethyl) disulfide; 3,3'-bis(tri-t-butoxyethyl) disulfide; 3,3'-bis(triisopropoxypropyl) disulfide; 3,3'-bis(trioctoxypropyl) disulfide; 2,2'-bis(2'-ethylhexoxysilylethyl) disulfide; 2,2'-bis(dimethoxy ethoxysilylethyl) disulfide; 3,3'-bis(methoxyethoxypropoxysilylpropyl) disulfide; 3,3'-bis(dimethoxymethylsilylpropyl) disulfide; 3,3'-bis(methoxy dimethylsilylpropyl) disulfide; 3,3'-bis(cyclohexoxy dimethylsilylpropyl) disulfide; 4,4'-bis(trimethoxysilylbutyl) disulfide; 3,3'-bis(trimethoxysilyl-3-methylpropyl) disulfide; 3,3'-bis(tripropoxysilyl-3-methylpropyl) disulfide; 3,3'-bis(dimethoxy methylsilyl-3-ethylpropyl) disulfide; 3,3'-bis(trimethoxysilyl-2-methylpropyl) disulfide; 3,3'-bis(dimethoxyphenylsilyl-2-methylpropyl) disulfide; 3,3'-bis(trimethoxysilylcyclohexyl) disulfide; 12,12'-bis(trimethoxysilyldodecyl) disulfide; 12,12'-bis(triethoxysilyldodecyl) disulfide; 18,18'-bis(trimethoxysilyloctadecyl) disulfide; 18,18'-bis(methoxydimethylsilyloctadecyl) disulfide; 2,2'-bis(trimethoxysilyl-2-methylethyl) disulfide; 2,2'-bis(triethoxysilyl-2-methylethyl) disulfide; 2,2'-bis(tripropoxysilyl-2-methylethyl) disulfide; 2,2'-bis(trioctoxysilyl-2-methylethyl) disulfide; 2,2'-bis(trimethoxysilyl-phenyl) disulfide; 2,2'-bis(triethoxysilyl-phenyl) disulfide; 1,1'-bis(trimethoxysilyl-tolyl)disulfide; 1,1'-bis(triethoxysilyl-tolyl)disulfide; 1,1'-bis(trimethoxysilyl-methyl tolyl) disulfide; 1,1'-bis(triethoxysilyl-methyl tolyl) disulfide; 2,2'-bis(trimethoxysilyl-ethyl phenyl) disulfide; 2,2'-bis(triethoxysilyl-ethyl phenyl) disulfide; 2,2'-bis(trimethoxysilyl-ethyl tolyl) disulfide; 2,2'-bis(triethoxysilyl-ethyl tolyl) disulfide; 3,3'-bis(trimethoxysilyl-propyl phenyl) disulfide; 3,3'-bis(triethoxysilyl-propyl phenyl) disulfide; 3,3'-bis(trimethoxysilyl-propyl tolyl) disulfide; and 3,3'-bis(triethoxysilyl-propyl tolyl) disulfide.

6. The process of claim 1 wherein said compound of formula II is selected from a group consisting of 2-mercaptoethyl trimethoxysilane, 3-mercaptopropyl trimethoxysilane; 2-mercaptopropyl triethoxysilane, 3-mercaptopropyl triethoxysilane, 2-mercaptoethyl tripropoxysilane, 2-mercaptoethyl tri sec.butoxysilane, 3-mercaptopropyl tri-t-butoxysilane, 3-mercaptopropyl triisopropoxysilane; 3-mercaptopropyl trioctoxysilane, 2-mercaptoethyl tri-2'-ethylhexoxysilane, 2-mercaptoethyl dimethoxy ethoxysilane, 3-mercaptopropyl methoxyethoxypropoxysilane, 3-mercaptopropyl dimethoxy methylsilane, 3-mercaptopropyl methoxy dimethylsilane, 3-mercaptopropyl cyclohexoxy dimethyl silane, 4-mercaptobutyl trimethoxysilane, 3-mercapto-3-methylpropyltrimethoxysilane, 3-mercapto-3-methylpropyl-tripropoxysilane, 3-mercapto-3-ethylpropyldimethoxy methylsilane, 3-mercapto-2-methylpropyl trimethoxysilane, 3-mercapto-2-methylpropyl dimethoxy phenylsilane, 3-mercaptocyclohexyl-trimethoxysilane, 12-mercaptododecyl trimethoxy silane, 12-mercaptododecyl triethoxy silane, 18-mercaptooctadecyl trimethoxysilane, 18-mercaptooctadecyl methoxydimethylsilane, 2-mercapto-2-methylethyltriethoxysilane, 2-mercapto-2-methylethyltripropoxysilane, 2-mercapto-2-methylethyltrioctoxysilane, 2-mercaptophenyl trimethoxysilane, 2-mercaptophenyl triethoxysilane; 1-mercaptotolyl trimethoxysilane; 1-mercaptotolyl triethoxysilane; 1-mercaptomethyltolyl trimethoxysilane; 1-mercaptomethyltolyl triethoxysilane; 2-mercaptoethylphenyl trimethoxysilane; 2-mercaptoethylphenyl triethoxysilane; 2-mercaptoethyltolyl trimethoxysilane; 2-mercaptoethyltolyl triethoxysilane; 3-mercaptopropylphenyl trimethoxysilane; 3-mercaptopropylphenyl triethoxysilane; 3-mercaptopropyltolyl trimethoxysilane; and 3-mercaptopropyltolyl triethoxysilane.

7. The process of claim 1 wherein said oxidation is in absence of water and in the presence of an organic solvent selected from the group consisting of chloroform, dichloromethane, carbon tetrachloride, hexane, heptane, cyclohexane, xylene, benzene, dichloroethylene, trichloroethylene, dioxane, diisopropyl ether, tetrahydrofuran and toluene.

8. The process of claim 1 wherein the oxidation reaction is conducted at a temperature ranging from 20° C. to 100° C.

9. The process of claim 8 wherein the oxidation reaction is conducted at a temperature ranging from 50° C. to 90° C.

10. The process of claim 1 wherein the oxidation reaction is conducted at a pressure ranging from 0.096 to 4.83 kg/cm^2.

* * * * *

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Search Results - Record(s) 1 through 2 of 2 returned.

☐ 1. Document ID: JP 04293948 A

L14: Entry 1 of 2 File: DWPI Oct 19, 1992

DERWENT-ACC-NO: 1992-394626
DERWENT-WEEK: 199248
COPYRIGHT 2001 DERWENT INFORMATION LTD

TITLE: Hot melt moulding compsn. for adhering to metals and plastic - comprises modified EVA copolymer, tackifier, and/or wax

PRIORITY-DATA: 1991JP-0059150 (March 22, 1991)

PATENT-FAMILY:

PUB-NO	PUB-DATE	LANGUAGE	PAGES	MAIN-IPC
JP 04293948 A	October 19, 1992	N/A	010	C08L023/26

INT-CL (IPC): C08L 23/26; C08L 29/04; C08L 31/04; C08L 57/02; C08L 91/06; C09J 129/04; C09J 131/04; C09J 191/06; C08L 23/26; C08L 25/10

Full | Title | Citation | Front | Review | Classification | Date | Reference | Claims | KWIC | Draw. Desc | Clip Img | Image

☐ 2. Document ID: JP 04292647 A

L14: Entry 2 of 2 File: DWPI Oct 16, 1992

DERWENT-ACC-NO: 1992-393977
DERWENT-WEEK: 199248
COPYRIGHT 2001 DERWENT INFORMATION LTD

TITLE: Hot melt compsn. useful for mfg. headlamps of motor cars - contains copolymer of hydrogenated styrene! with butadiene! and/or isoprene!, EVA and tackifier, wax and/or plasticiser

PRIORITY-DATA: 1991JP-0059149 (March 22, 1991)

PATENT-FAMILY:

PUB-NO	PUB-DATE	LANGUAGE	PAGES	MAIN-IPC
JP 04292647 A	October 16, 1992	N/A	009	C08L025/10

INT-CL (IPC): C08L 23/26; C08L 25/10; C08L 31/04; C08L 91/06; C08L 93/00; C09J 125/10

Full | Title | Citation | Front | Review | Classification | Date | Reference | Claims | KWIC | Draw. Desc | Image

captopropyl triethoxysilane were slowly added. During the period of addition, the reaction mixture was vigorously stirred. When the reaction reached about 90° C., 500 ml of chloroform were added. Stirring continued for an additional one hour. The solid was filtered and washed with chloroform. The organic phase was filtered on charcoal to eliminate all the MnO_2. The solvent was evaporated under reduced pressure to obtain 470 grams (0.99 mole) of product. The product was subjected to gas chromatographic analysis. The purity was determined to exceed 95 percent of desired product.

EXAMPLE 5

Preparation of bis(triethoxysilylpropyldisulfide)

In a 2-liter beaker, 2,740 grams (31.5 mole) of MnO_2 were placed. Thereafter, 500 grams (2.1 mole) of 3-mercaptopropyl triethoxysilane were slowly added. During the period of addition, the reaction mixture was vigorously stirred. When the reaction reached about 90° C., 500 ml of chloroform were added. Stirring continued for an additional one hour. The solid was filtered and washed with chloroform. The organic phase was filtered on charcoal to eliminate all the MnO_2. The solvent was evaporated under reduced pressure to obtain 470 grams (0.99 mole) of product. The product was subjected to gas chromatographic analysis. The purity was determined to exceed 95 percent of desired product.

EXAMPLE 6

Preparation of bis(triethoxysilylpropyldisulfide)

In a 2-liter beaker, 1,827 grams (21 mole) of MnO_2 were placed. Thereafter, 500 grams (2.1 mole) of 3-mercaptopropyl triethoxysilane were slowly added. During the period of addition, the reaction mixture was vigorously stirred. When the reaction reached about 90° C., 500 ml of chloroform were added. Stirring continued for an additional one hour. The solid was filtered and washed with chloroform. The organic phase was filtered on charcoal to eliminate all the MnO_2. The solvent was evaporated under reduced pressure to obtain 470 grams (0.99 mole) of product. The product was subjected to gas chromatographic analysis. The purity was determined to exceed 95 percent of desired product.

EXAMPLE 7

Preparation of bis(triethoxysilylpropyldisulfide)

In a 2-liter beaker, 913.5 grams (10.5 mole) of MnO_2 were placed. Thereafter, 500 grams (2.1 mole) of 3-mercaptopropyl triethoxysilane were slowly added. During the period of addition, the reaction mixture was vigorously stirred. When the reaction reached about 90° C., 500 ml of chloroform were added. Stirring continued for an additional one hour. The solid was filtered and washed with chloroform. The organic phase was filtered on charcoal to eliminate all the MnO_2. The solvent was evaporated under reduced pressure to obtain 470 grams (0.99 mole) of product. The product was subjected to gas chromatographic analysis. The purity was determined to exceed 95 percent of desired product.

EXAMPLE 8

Preparation of bis(triethoxysilylpropyldisulfide)

In a 2-liter beaker, 365.4 grams (4.2 mole) of MnO_2 were placed. Thereafter, 500 grams (2.1 mole) of 3-mercaptopropyl triethoxysilane were slowly added. During the period of addition, the reaction mixture was vigorously stirred. When the reaction reached about 90° C., 500 ml of chloroform were added. Stirring continued for an additional one hour. The solid was filtered and washed with chloroform. The organic phase was filtered on charcoal to eliminate all the MnO_2. The solvent was evaporated under reduced pressure to obtain 470 grams (0.99 mole) of product. The product was subjected to gas chromatographic analysis. The purity was determined to exceed 95 percent of desired product.

EXAMPLE 9

Preparation of bis(triethoxysilylpropyldisulfide)

In a 2-liter beaker, 320 grams (3.68 mole) of MnO_2 were placed. Thereafter, 500 grams (2.1 mole) of 3-mercaptopropyl triethoxysilane were slowly added. During the period of addition, the reaction mixture was vigorously stirred. When the reaction reached about 90° C., 500 ml of dichloromethane were added. Stirring continued for an additional one hour. The solid was filtered and washed with dichloromethane. The organic phase was filtered on charcoal to eliminate all the MnO_2. The solvent was evaporated under reduced pressure to obtain 470 grams (0.99 mole) of product. The product was subjected to gas chromatographic analysis. The purity was determined to exceed 95 percent of desired product.

EXAMPLE 10

Preparation of bis(triethoxysilylpropyldisulfide)

In a 2-liter beaker, 320 grams (3.68 mole) of MnO_2 were placed. Thereafter, 500 grams (2.1 mole) of 3-mercaptopropyl triethoxysilane were slowly added. During the period of addition, the reaction mixture was vigorously stirred. When the reaction reached about 90° C., 500 ml of carbon tetrachloride were added. Stirring continued for an additional one hour. The solid was filtered and washed with carbon tetrachloride. The organic phase was filtered on charcoal to eliminate all the MnO_2. The solvent was evaporated under reduced pressure to obtain 470 grams (0.99 mole) of product. The product was subjected to gas chromatographic analysis. The purity was determined to exceed 95 percent of desired product.

EXAMPLE 11

Preparation of bis(triethoxysilylpropyldisulfide)

In a 2-liter beaker, 320 grams (3.68 mole) of MnO_2 were placed. Thereafter, 500 grams (2.1 mole) of 3-mercaptopropyl triethoxysilane were slowly added. During the period of addition, the reaction mixture was vigorously stirred. When the reaction reached about 90° C., 500 ml of dichloroethylene were added. Stirring continued for an additional one hour. The solid was filtered and washed with dichloroethylene. The organic phase was filtered on charcoal to eliminate all the MnO_2. The solvent was evaporated under reduced pressure to obtain 470 grams (0.99 mole) of product. The product was subjected to gas chromatographic analysis. The purity

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Search Results - Record(s) 1 through 3 of 3 returned.

☐ 1. Document ID: US 6218017 B1

L6: Entry 1 of 3 File: USPT Apr 17, 2001

US-PAT-NO: 6218017
DOCUMENT-IDENTIFIER: US 6218017 B1

TITLE: Laminated structure, covering structure and pouch

DATE-ISSUED: April 17, 2001

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Yamashita; Rikiya	Shinjuku-ku	N/A	N/A	JPX
Matsuzaki; Hiroshi	Shinjuku-ku	N/A	N/A	JPX
Yamazaki; Takuya	Shinjuku-ku	N/A	N/A	JPX

US-CL-CURRENT: 428/424.2; 428/458, 428/461, 428/480, 428/483, 428/500

Full | Title | Citation | Front | Review | Classification | Date | Reference | Claims | KWIC | Draw. Desc | Image

☐ 2. Document ID: JP 11199840 A

L6: Entry 2 of 3 File: DWPI Jul 27, 1999

DERWENT-ACC-NO: 1999-474234
DERWENT-WEEK: 199944
COPYRIGHT 2001 DERWENT INFORMATION LTD

TITLE: Base material for pressure sensitive adhesive tape - used for pressure sensitive adhesive tape and parting tape-bearing pressure sensitive adhesive tape

PRIORITY-DATA: 1998JP-0006759 (January 16, 1998)

PATENT-FAMILY:

PUB-NO	PUB-DATE	LANGUAGE	PAGES	MAIN-IPC
JP 11199840 A	July 27, 1999	N/A	007	C09J007/02

INT-CL (IPC): B32B 27/00; B32B 27/28; B32B 27/32; C09J 7/02; H01L 21/301

Full | Title | Citation | Front | Review | Classification | Date | Reference | Claims | KWIC | Draw. Desc | Image

☐ 3. Document ID: JP 07169307 A

L6: Entry 3 of 3 File: DWPI Jul 4, 1995

PROCESS FOR THE PREPARATION OF ORGANOSILICON DISULFIDE COMPOUNDS

BACKGROUND OF THE INVENTION

The present invention relates to a process for the preparation of organo silicon disulfide compounds. Organo silicon disulfides are known adhesion promoters in sulfur-vulcanizable rubber mixtures reinforced with inorganic materials such as glass SiO_2, aluminosilicates and carbon black. For example, in GB 1,484,909, there is disclosed a process for the preparation of organo trialkoxysilane disulfides. In accordance with the teachings of this reference, mercaptopropyl trimethoxy silane or mercaptopropyl triethoxy silane is reacted with sulfuryl chloride in an inert solvent at temperatures of from 0° to 100°. The disulfide is then obtained by fractional distillation. The yields of desired product range in the neighborhood of 63 to 65 percent of theoretical.

U.S. Pat. No. 3,842,111 discloses a method for the preparation of organosilicon disulfide compounds by oxidizing mercaptoalkoxysilanes. Representative oxidizing agents include oxygen, chlorine, halogens of atomic weight 35 to 127, nitric oxide, sulfuryl chloride and sulfoxides.

Generally speaking, organosilicon disulfide compounds are very expensive and, with the increasing interest in silica-reinforced vulcanizable rubber, more cost-efficient methods of preparing these compounds are needed.

SUMMARY OF THE INVENTION

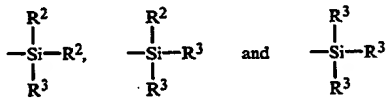
The present invention relates to a process for the preparation of a organosilicon disulfide compounds of the formula

$$Z—R^1—S_2—R^1—Z \quad \text{I}$$

comprising oxidizing a compound of the formula

$$Z—R^1—SH \quad \text{II}$$

in the presence of manganese dioxide, wherein Z is selected from the group consisting of



wherein R^2 may be the same or different and is independently selected from the group consisting of an alkyl group having 1 to 4 carbons and phenyl; R^3 may be the same or different and is independently selected from the group consisting of alkyl groups having 1 to 4 carbon atoms, phenyl, alkoxy groups having 1 to 8 carbon atoms and cycloalkoxy groups with 5 to 8 carbon atoms; and R^1 is selected from the group consisting of a substituted or unsubstituted alkylene group having a total of 1 to 18 carbon atoms and a substituted or unsubstituted arylene group having a total of 6 to 12 carbon atoms.

DETAILED DESCRIPTION OF THE INVENTION

The present inventi n relates to a process for the preparation of rganosilicon disulfide compounds. Representative organosilicon disulfide compounds of formula I which may be prepared in accordance with the present invention include 2,2'-bis(trimethoxysilylethyl) disulfide; 3,3'-bis(trimethoxysilylpropyl) disulfide; 3,3'-bis(triethoxysilylpropyl) disulfide; 2,2'-bis(triethoxysilylpropyl) disulfide; 3,3'-bis(triethoxysilylpropyl) disulfide; 2,2'-bis(tripropoxysilylethyl) disulfide; 2,2'-bis(trisec.butoxysilylethyl) disulfide; 3,3'-bis(tri-t-butoxyethyl) disulfide; 3,3'-bis(triisopropoxypropyl) disulfide; 3,3'-bis(trioctoxypropyl) disulfide; 2,2'-bis(2'-ethylhexoxysilylethyl) disulfide; 2,2'-bis(dimethoxy ethoxysilylethyl) disulfide; 3,3'-bis(methoxyethoxypropoxysilylpropyl) disulfide; 3,3'-bis(dimethoxymethylsilylpropyl) disulfide; 3,3'-bis(methoxy dimethylsilylpropyl) disulfide; 3,3'-bis(cyclohexoxy dimethylsilylpropyl) disulfide; 4,4'-bis(trimethoxysilylbutyl) disulfide; 3,3'-bis(trimethoxysilyl-3-methylpropyl) disulfide; 3,3'-bis(tripropoxysilyl-3-methylpropyl) disulfide; 3,3'-bis(dimethoxy methylsilyl-3-ethylpropyl) disulfide; 3,3'-bis(trimethoxysilyl-2-methylpropyl) disulfide; 3,3'-bis(dimethoxyphenylsilyl-2-methylpropyl) disulfide; 3,3'-bis(trimethoxysilylcyclohexyl) disulfide; 12,12'-bis(trimethoxysilyldodecyl) disulfide; 12,12'-bis(triethoxysilyldodecyl) disulfide; 18,18'-bis(trimethoxysilyloctadecyl) disulfide; 18,18'-bis(methoxydimethylsilyloctadecyl) disulfide; 2,2'-bis(trimethoxysilyl-2-methylethyl) disulfide; 2,2'-bis(triethoxysilyl-2-methylethyl) disulfide; 2,2'-bis(tripropoxysilyl-2-methylethyl) disulfide; 2,2'-bis(trioctoxysilyl-2-methylethyl) disulfide; 2,2'-bis(trimethoxysilyl-phenyl) disulfide; 2,2'-bis(triethoxysilyl-phenyl) disulfide; 1,1'-bis(trimethoxysilyl-tolyl)disulfide; 1,1'-bis(triethoxysilyl-tolyl)disulfide; 1,1'-bis(trimethoxysilyl-methyl tolyl) disulfide; 1,1'-bis(triethoxysilyl-methyl tolyl) disulfide; 2,2'-bis(trimethoxysilyl-ethyl phenyl) disulfide; 2,2'-bis(triethoxysilyl-ethyl phenyl) disulfide; 2,2'-bis(trimethoxysilyl-ethyl tolyl) disulfide; 2,2'-bis(triethoxysilyl-ethyl tolyl) disulfide; 3,3'-bis(trimethoxysilyl-propyl phenyl) disulfide; 3,3'-bis(triethoxysilyl-propyl phenyl) disulfide; 3,3'-bis(trimethoxysilyl-propyl tolyl) disulfide; and 3,3'-bis(triethoxysilyl-propyl tolyl) disulfide.

With reference to formula I, preferably R^1 is a alkylene group having 1 to 3 carbon atoms

Z is

ps and R^3 is an alkoxy group having from 1 to 2 carbon atoms.

The desired products are prepared by oxidizing a compound of formula II. Representative examples of compounds of formula II include 2-mercaptoethyl trimethoxysilane, 3-mercaptopropyl trimethoxysilane, 3-mercaptopropyl triethoxysilane, 2-mercaptopropyl triethoxysilane, 2-mercaptoethyl tripropoxysilane, 2-mercaptoethyl tri sec.butoxysilane, 3-mercaptopropyl tri-t-butoxysilane, 3-mercaptopropyl triisopropoxysilane; 3-mercaptopropyl trioctoxysilane, 2-mercaptoethyl tri-2'-ethylhexoxysilane, 2-mercaptoethyl dimethoxy ethoxysilane, 3-mercaptopropyl methoxyethoxypropoxysilane, 3-mercaptopropyl dimethoxy methylsilane, 3-mercaptopropyl methoxy dimethylsilane, 3-mercaptopropyl cyclohexoxy dimethyl silane, 4-mercaptobutyl trimethoxysilane, 3-mercapto-3-methylpropyl-trimethoxysilane, 3-mercapto-3-methylpropyl-tripropoxysilane, 3-mercapto-3-ethylpropyl-dimethoxy

DERWENT-ACC-NO: 1995-266895
DERWENT-WEEK: 199535

TITLE: Sealing material for vehicle lamps - comprises hydrogenated styrene! polymer, pressure-sensitive adhesive and plasticiser

PRIORITY-DATA: 1993JP-0316432 (December 16, 1993)

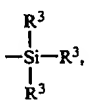
PATENT-FAMILY:

PUB-NO	PUB-DATE	LANGUAGE	PAGES	MAIN-IPC
JP 07169307 A	July 4, 1995	N/A	009	F21Q001/00

INT-CL (IPC): C08F 12/08; F21Q 1/00

methylsilane, 3-mercapto-2-methylpropyl trimethoxysilane, 3-mercapto-2-methylpropyl dimethoxy phenylsilane, 3-mercaptocyclohexyl-trimethoxysilane, 12-mercaptododecyl trimethoxy silane, 12-mercaptododecyl triethoxy silane, 18-mercaptooctadecyl trimethoxysilane, 18-mercaptooctadecyl methoxydimethylsilane, 2-mercapto-2-methylethyltriethoxysilane, 2-mercapto-2-methylethyltripropoxysilane, 2-mercapto-2-methylethyltrioctoxysilane, 2-mercaptophenyl trimethoxysilane, 2-mercaptophenyl triethoxysilane; 1-mercaptotolyl trimethoxysilane; 1-mercaptotolyl triethoxysilane; 1-mercaptomethyltolyl trimethoxysilane; 1-mercaptomethyltolyl triethoxysilane; 2-mercaptoethylphenyl trimethoxysilane; 2-mercaptoethylphenyl triethoxysilane; 2-mercaptoethyltolyl trimethoxysilane; 2-mercaptoethyltolyl triethoxysilane; 3-mercaptopropylphenyl trimethoxysilane; 3-mercaptopropylphenyl triethoxysilane; 3-mercaptopropyltolyl trimethoxysilane; and 3-mercaptopropyltolyl triethoxysilane.

With reference to formula II, preferably Z is



R^3 is an alkoxy group having from 1 to 2 carbon atoms and R^1 is an alkylene group having 2 to 3 carbon atoms.

The mercapto compound of formula II is oxidized in the presence of manganese dioxide. Whereas commercially available manganese dioxide may be used, it is preferred to use freshly prepared manganese dioxide. Manganese dioxide may be prepared by adding an aqueous suspension of $MnSO_4 \cdot H_2O$ and sodium hydroxide to aqueous solution of $KMnO_4$. The mixture is allowed to react, the precipitate is isolated, washed and then dried. To increase the surface area of the manganese dioxide product, it is preferred to grind the manganese dioxide before use. Another way to significantly increase the active MnO_2 surface is to deposit the oxide onto an inert carrier like silica, alumosilicates, charcoal and the like. It has also been discovered that, after the manganese dioxide has been used in the process of the present invention, the isolated manganese dioxide is preferably regenerated. The manganese dioxide may be regenerated by drying the manganese dioxide at 120° C. The manganese dioxide may also be regenerated by adding the manganese dioxide to a boiling mixture of water and Norvanol ®. Norvanol ® is a commercially available mixture of 83 percent ethanol, 3 percent ether and 14 percent water. After a suitable time, such as 2 hours, the manganese dioxide is isolated, washed and dried at 120° C.

The process of the present invention involves oxidizing the compound of formula II in the presence of manganese dioxide. The molar ratio of the compound of formula II to manganese dioxide may range from 1:0.025 to 1:45. Preferably, the molar ratio ranges from 1:0.5 to 1:25 with a range of from 1:4 to 1:1.7 being particularly preferred.

The oxidation reaction should be conducted in the absence of water because the presence of a siloxane moiety may be hydrolysed by contact with water.

The oxidation reaction of the present invention may be conducted in the presence of an organic solvent. Suitable solvents which may be used include chloroform, dichloromethane, carbon tetrachloride, hexane, heptane, cyclohexane, xylene, benzene, dichloroethylene, trichloroethylene, dioxane, diisopropyl ether, tetrahydrofuran and toluene. As indicated above, care should be exercised to avoid the presence of water during the reaction. Therefore, none of the above solvent should contain any appreciable levels of water. Preferably, the organic solvent is chloroform, heptane, cyclohexane and toluene.

The oxidation reaction may be conducted over a variety of temperatures. Generally speaking, the oxidation reaction is conducted in a temperature ranging from 20° C. to 100° C. Preferably, the reaction is conducted at a temperature ranging from 50° C. to 90° C.

The process of the present invention may be conducted at a variety of pressures. Generally speaking, however, the oxidation reaction is conducted at a pressure ranging from 0.096 to 4.83 kg/cm^2.

EXAMPLE 1

Synthesis of Manganese Dioxide

Into the reaction vessel was added an aqueous solution of 31.6 grams (0.2 mole) of $KMnO_4$ (200 ml of water). The solution was vigorously stirred to which was added a suspension of 50.7 grams (0.3 mole) of $MnSO_4 \cdot H_2O$, 20 grams (0.2 mole) of NaOH) and 200 ml of water. Stirring was continued for one hour. The precipitate was isolated, washed with water and dried at 120° C. The dried product was ground to a fine powder.

EXAMPLE 2

Preparation of bis(triethoxysilylpropyldisulfide)

In a 2-liter beaker, 320 grams (3.68 mole) of MnO_2 were placed. Thereafter, 500 grams (2.1 mole) of 3-mercaptopropyl triethoxysilane were slowly added. During the period of addition, the reaction mixture was vigorously stirred. When the reaction reached about 90° C., 500 ml of chloroform were added. Stirring continued for an additional one hour. The solid was filtered and washed with chloroform. The organic phase was filtered on charcoal to eliminate all the MnO_2. The solvent was evaporated under reduced pressure to obtain 470 grams (0.99 mole) of product. The product was subjected to gas chromatographic graphic analysis. The purity was determined to exceed 95 percent of desired product.

EXAMPLE 3

Preparation of bis(triethoxysilylpropyldisulfide)

In a 2-liter beaker, 4,567 grams (52.5 mole) of MnO_2 were placed. Thereafter, 500 grams (2.1 mole) of 3-mercaptopropyl triethoxysilane were slowly added. During the period of addition, the reaction mixture was vigorously stirred. When the reaction reached about 90° C., 500 ml of chloroform were added. Stirring continued for an additional one hour. The solid was filtered and washed with chloroform. The organic phase was filtered on charcoal to eliminate all the MnO_2. The solvent was evaporated under reduced pressure to obtain 470 grams (0.99 mole) of product. The product was subjected to gas chromatographic analysis. The purity was determined to exceed 95 percent of desired product.

EXAMPLE 4

Preparation of bis(triethoxysilylpropyldisulfide)

In a 2-liter beaker, 3,654 grams (42 mole) of MnC_2 were placed. Thereafter, 500 grams (2.1 mole) of 3-mer-

DB Name	Query	Hit Count	Set Name
USPT,PGPB,JPAB,EPAB,DWPI,TDBD	l9 and (l11 or l12) and l13 and l14	0	L17
USPT,PGPB,JPAB,EPAB,DWPI,TDBD	l9 and (l11 or l12) and l13	1	L16
USPT,PGPB,JPAB,EPAB,DWPI,TDBD	l9 and (l11 or l12)	8	L15
USPT,PGPB,JPAB,EPAB,DWPI,TDBD	liquid paraffin	12284	L14
USPT,PGPB,JPAB,EPAB,DWPI,TDBD	terpene resin	4194	L13
USPT,PGPB,JPAB,EPAB,DWPI,TDBD	hydrogenated styrene isoprene copolymer	46	L12
USPT,PGPB,JPAB,EPAB,DWPI,TDBD	hydrogenated styrene butadiene copolymer	162	L11
USPT,PGPB,JPAB,EPAB,DWPI,TDBD	l6 and l9	3	L10
USPT,PGPB,JPAB,EPAB,DWPI,TDBD	sealant	53604	L9
USPT,PGPB,JPAB,EPAB,DWPI,TDBD	L1 and L2 and l3 or l4 [ab]	8	L8
USPT,PGPB,JPAB,EPAB,DWPI,TDBD	L1 and L2 and l3 or l4 [ti]	0	L7
USPT,PGPB,JPAB,EPAB,DWPI,TDBD	L1 and L2 and l3 or l4	35	L6
USPT,PGPB,JPAB,EPAB,DWPI,TDBD	L1 AND L2 and l3 or l4	35	L5
USPT,PGPB,JPAB,EPAB,DWPI,TDBD	HV-300	35	L4
USPT,PGPB,JPAB,EPAB,DWPI,TDBD	HV-100	16	L3
USPT,PGPB,JPAB,EPAB,DWPI,TDBD	ESCOREZ 5320	59	L2
USPT,PGPB,JPAB,EPAB,DWPI,TDBD	KRATON G-1652	148	L1

resin, thus the wax prevents aggregation of the pigment particles which is one cause of streaking and poor coloring of plastic resin using previously known colorants. The pigmented plastic resin or rubber composition is produced by mixing the colorant pr duced in accordance with the method described above with the normal plastic resin or rubber to produce a brilliant colored end composition which is made possible because of the ability of the wax-coated pigment to be wetted by the plastic resin or rubber during mixing with the new colorant. The high pigment loading is made possible by using a colorant produced in accordance with the present invention.

In accordance with the invention, a hydroxystearate wax is used as the coating agent. The preferred hydroxystearate wax is glyceryl-tris-12-hydroxystearate: C_3H_5-$(OOCC_{17}H_{34}OH)_3$. Other examples of hydroxystearate waxes are methyl hydroxystearate, propylene glycol mono-hydroxystearate, glyceryl mono-hydroxystearate, ethylene glycol mono-hydroxystearate and stearyl-12-hydroxystearate. Glyceryl-tris-12-hydroxystearate is a hard, brittle wax-like solid with a melting point temperature of 86° C.–88° C. (about 188° F.). The listed hydroxystearate waxes have melting point temperatures in the range of 52° C.–88° C. which is substantially below the normal boiling point temperature of water. These hydroxystearate waxes are insoluble at room temperature in most organic solvents, but are highly compatible with plastic resins and waxes. These waxes have sharp melting points and minimum shrinkage and are usable in food packaging. This material assists in mold release when compounded with a plastic resin and adds in blending of some plastic materials. These waxes are saturated ricinoleate esters which have medium range melting wax-like solids that exhibit oxidation stability and oil and water resistance.

The term pigment means substances which are generally considered insoluble in the vehicle, and pigments generally have the property of light refractivity. (Dyes are considered soluble and generally have only the property of light absorption.) Phosphorescent, luminescent, fluorescent, metalescent, and pearlescent materials fit within the term pigment, as used herein and in the art. The pigment must be in particle form and should have a mean particle size between about 0.1 and about 100 microns and preferably between about 0.2 and about 50 microns. The most preferred mean particle size for organic pigments is about 0.2 micron. The most preferred particle size for inorganic pigments is about 50 microns.

Examples of organic and inorganic pigments which can be used in this invention are iron blue, zinc oxide, titanium dioxide, chrome yellow, carbon black, chrome orange, chrome green, zinc chromate, red lead, lethol red, the lakes, azo type toners, phthalocyanines, aluminum hydrates, lakes, iron oxide, white lead, extenders, phosphotungstic acid toners, titanium-containing pigments, sulfur-containing pigments, extenders, calcium carbonate, aluminum oxide, lithopane, ultraphone, lead chromate, cadmium sulfide, cadmium selenide, barium sulfate, azo pigments, anthraquinone and vat pigments, phthalocyanine pigments, acrylamino yellow, magnesium oxide, chrome red, antimony oxide, zinc sulfide, magnesium fluoride and ground barytes. Benzoid pigments are useful and examples are toners and lakes. Examples of benzoid toners are yellow toners, e.g., benzoid yellows and Hansa yellows; orange toners, e.g. vat orange 3; red toners, .g., napthol reds; violet toners; blue toners; green toners; brown toners; and black toners. Examples of bezoid lakes are yellow lakes, e.g., acid yellow 2; orange lakes; red lakes; vi let lakes; blue lakes; e.g. acid blue 93; green lakes; brown lakes; and black lakes, e.g., natural black 3. Metallic pigments can be used, and examples are aluminum flakes. Mixtures of pigments can be used.

In the present invention, the pigment particles may be presscake particles. These are produced in a non-agglomerated condition by a process which results in 30%–50% combined water. By using the present invention, the presscake particles are simultaneously dehydrated and coated with a hydroxystearate wax. The equipment used is open to the atmosphere which allows for nearly complete extraction of entrapped moisture which would be generally impossible in closed equipment such as extruders or batch mixers. High shear forces applied to the mixture also assist in positive expulsion of entrapped moisture. In prior systems, the standard presscake form of pigments required an oven drying step to expell moisture. This required additional energy and caused agglomeration of pigment particles. Consequently, the agglomerated particles had to be ground which was difficult and required special equipment. If the dried particles were not immediately processed, they again absorbed moisture. For these reasons, moisture content of prior colorants often was at least 10% by weight. The present invention uses the hydrated, non-agglomerated pigment particles without prior drying or other processing. Of course, dried and/or ground pigment particles can be used in the present invention with the advantage that absorbed moisture is positively expelled during the process without special equipment or high energy requirements.

Any thermoplastic material can be formulated with the coated pigments of this invention. Examples of thermoplastics with which the pigment dispersions can be used are: the ABS resins prepared from acrylonitrile, butadiene and styrene; blends of ABS resins with other thermoplastics, such as, polyvinylchloride; resins prepared from acrylonitrile, butadiene, styrene and alpha methyl styrene; resins prepared from butadiene, styrene and methacrylic acid; resins prepared from acrylonitrile, butadiene, styrene and methyl methacrylate acetal copolymers; acetal resins; acrylic resins and modified acrylic resins, such as, polymethyl methacrylate, copolymers of styrene and methyl methacrylate, copolymers of methyl methacrylate and alpha methyl styrene; the cellulosic plastics, such as, cellulose acetate plastics, cellulose acetate butyrate plastics, cellulose propionate plastics, ethyl cellulose plastics and cellulose nitrate plastics; mixtures of ethyl cellulose plastics and cellulose acetate butyrate; chorinated polyether; the fluoroplastics; such as, polytetrafluoroethylene, polyvinylidene fluoride, the fluorinated ethylene-propylene plastics and the chlorotrifluoroethylene plastics; the phenoxy resins; the polybutadiene-type resins, such as, butadiene-styrene copolymer and polybutadiene; the polycarbonates; the polyethylene resins, such as, low-density polyethylene; copolymers of polyethylene with other materials; chlorinated polyethylenes; chlorosulfonated polyethylenes; ethylene vinyl acetate copolymer; ethylene acrylate copolymer; polyphenylene oxide; the polypropylenes; the polysulfones; the polystyrenes; styrene copolymers, such as, styrene-methyl methacrylate copolymer; and vinyl polymers and copolymers, such as, polyvinyl chloride, copolymer of vinyl chloride and vinyl acetate, copolymer of vinyl chloride,

WEST

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Search Results - Record(s) 1 through 8 of 8 returned.

☐ 1. Document ID: US 6218017 B1

L15: Entry 1 of 8 File: USPT Apr 17, 2001

US-PAT-NO: 6218017
DOCUMENT-IDENTIFIER: US 6218017 B1

TITLE: Laminated structure, covering structure and pouch

DATE-ISSUED: April 17, 2001

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Yamashita; Rikiya	Shinjuku-ku	N/A	N/A	JPX
Matsuzaki; Hiroshi	Shinjuku-ku	N/A	N/A	JPX
Yamazaki; Takuya	Shinjuku-ku	N/A	N/A	JPX

US-CL-CURRENT: 428/424.2; 428/458, 428/461, 428/480, 428/483, 428/500

Full | Title | Citation | Front | Review | Classification | Date | Reference | Claims | KWIC | Draw Desc | Image

☐ 2. Document ID: US 6089646 A

L15: Entry 2 of 8 File: USPT Jul 18, 2000

US-PAT-NO: 6089646
DOCUMENT-IDENTIFIER: US 6089646 A

TITLE: Vehicular window assembly

DATE-ISSUED: July 18, 2000

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Xu; Qihua	Holland	MI	N/A	N/A
Nestell; David E.	Spring Lake	MI	N/A	N/A

US-CL-CURRENT: 296/146.15; 296/93, 52/204.591

Full | Title | Citation | Front | Review | Classification | Date | Reference | Claims | KWIC | Draw Desc | Image

☐ 3. Document ID: US 6086138 A

L15: Entry 3 of 8 File: USPT Jul 11, 2000

vinyl acetate and vinyl alcoh l, copolymer of vinyl chloride and vinylidene chloride, and polyvinyldichloride.

Reinforced thermoplastics can be used. The reinforcing is normally d ne with glass fibers, fibrous asbestos, metal fibers, refractory fibers, and other fibers.

Various fillers can be used in the thermoplastics compositions. Examples of the fillers are calcium carbonate, carbon black, clay, asbestos, mica, talc, barium sulfate, magnesium oxide, barium carbonate, ground glass, and metal powders. It is noted that several of the fillers are also listed as useful pigments. A material can be used as a pigment and a filler, but when it is used as a pigment it must have been treated with a hydroxystearate wax.

The thermoplastic compositions can contain other conventional materials, such as, plasticizers, stabilizers, flame retardants, UV absorbers, antistatic additives, etc.

The coated pigment can contain from about 10 to about 90 percent by weight of pigment and from about 90 to about 10 percent by weight of hydroxystearate wax, although the preferred amount of pigment is 30 to 75 percent by weight and the preferred amount of wax is 70 to 25 percent by weight. Between about 0.1 and about 10 parts by weight of the coated pigment can be formulated 100 parts by weight of the thermoplastic, although the preferred amount of coated pigment is 0.5 to 3.0 parts by weight per 100 parts by weight of the thermoplastic.

The coated pigments include an individual pigment encased in the vehicle or several individual pigments encased in the vehicle, but there is no agglomeration of the pigments in the vehicle. The wax solidifies around the encapsulated pigment particles. There is development of essentially the full color potential of the pigments. This is achieved through the superior wetting action of the hydroxystearate vehicle. Also, extremely high pigment loadings are possible with the coated pigments, for example, 100 parts of coated pigment to one part of resin.

The coated pigment is dustless, which is extremely important in a commerical sense. The coated pigment are not drawn together by standing or due to vibration, the latter is very important when automated coloring systems are used.

The coated pigment particles (after being sized) are formulated and formed with uncolored thermoplastic material particles in or by some low-shear compounding means, such as, an injection molder, rotational casting means, blow molding machinery, extruder, Banbury-type mechanical mixer and compounding rolls. (High shear compounding means can be used, but is not necessary or preferred.) The final product can have any shape, such as, film, sheet, rod, filament, cubical, spherical, etc. The final product can even be formed. Thus the dry coloring of formed thermoplastic materials is achieved without the use of high shear as is necessary in the prior art methods and with prior art pigments.

Referring now to the Figures, FIG. 1 shows an apparatus A used in producing the desired colorant flakes from pigment particles which may have upwards of 50% by weight of entrapped moisture. This apparatus is a somewhat standard three roll mill having horizontally aligned, adjacent rolls 10, 12, 14. In accordance with the invention, rolls 10, 12 are used for initial heating and mixing f the pigment particles and hydroxystearate particles. To produce high shear and desired mixing, the rolls 10, 12 are rotated at a different speed. The faster roll 12 is operated at a speed of 3 X which is three times the speed X of roll 10. In practice, the rolls are chromium plated steel having a diameter of 10 inches and a length of 30 inches and the speed X is 50 RPM. The outlet or exit roll 14 is driven at a speed of 9 X which is three times the speed of roll 12. These rolls are situated in an open receptacle 20 which closes the opposite ends of the rolls 10, 12 and 14 to cause the material to travel in the path illustrated in FIG. 1. In the area 30 which is above the nip between rolls 10, 12 the pigment particles and wax particles are heated and mixed. In practice, rolls 10, 12 are heated to a temperature in the range of 250° F.–450° F. Preferably, the rolls are approximately 400° F. This produces a rapid heating of the particle mixture in the area 30 which causes a rapid expulsion of moisture from the pigment particles and a simultaneous coating of the particles by melting of the wax within the area 30. This produces a turning and kneading action of the particles. The melted wax, in a somewhat lower shear type of mixing action, is preparatory to the mixture of molten wax and pigment particles in high shear at the nip area 32 between rolls 10, 12. In this nip area, the mixture is subjected to a high shear mechanical action which assists in the final moisture expulsion from the pigment particles. This high shear action causes a fine, even dispersion of pigment particles within the heated wax matrix. The general mixing and compressing action between rolls 10, 12 is schematically illustrated in FIG. 2. The pigment and molten wax mixture clings to the high speed roll in accordance with normal practice and passes around the roll 12 to the nip between roll 12 and roll 14. This produces an additional high shear mixing action. When contacting roll 14, which is cooled to a temperature of a general range of minus 10° F. to 100° F., the wax matrix solidifies into a solid mass which is carried over roll 14 to an exit scraper or chipper 40, which chips the material from roll 14 into discrete flakes of coated pigment particles. The nip spacing between rolls 10 and 12 is in the general range of approximately 0.005–0.010 inches. Thus, the flexible mixture on roll 12 is generally in the range of 0.005–0.10 inches in thickness. This pigment is retained on roll 14 so that a very fine solidified layer appears on roll 14 at the chipper 40. This thin layer is rapidly cooled to the temperature of roll 14 prior to reaching the chipper. Thus, since the hydroxystearate wax is brittle and hard at approximately room temperature, the chipper produces fractured flakes of colorant which may include several wax-encapsulated pigment particles. The apparatus A with heated and cooled rolls is an available ink mill and is somewhat inexpensive. It is noted that prior drying and grinding of the pigment particles is not required. In addition, complicated and expensive extruders and mixing devices are not necessary. In using the apparatus, the high shear mixing and heating of the material is effected conveniently and quite rapidly. The wax particles and pigment particles are loaded into the area above nip 32 in bulk in proportion to that desired in the final product. Modifications could be made in the apparatus without departing from the apparatus aspect of the present invention as long as the operating characteristics schematically illustrated in FIGS. 1 and 2 are utilized.

EXAMPLE 1

1000 g of Cadmium orange presscake pigment was admixed with 500 g of glyceryl-tris-12-hydroxystearate. The resulting mixture was placed in a three roll mill as shown in FIG. 1 in which the front two rolls 10, 12

US-PAT-NO: 6086138
DOCUMENT-IDENTIFIER: US 6086138 A

TITLE: Vehicular window assembly

DATE-ISSUED: July 11, 2000

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Xu; Qihua	Holland	MI	N/A	N/A
Nestell; David E.	Spring Lake	MI	N/A	N/A

US-CL-CURRENT: 296/146.15; 296/93, 52/204.591

Full | Title | Citation | Front | Review | Classification | Date | Reference | Claims | KWIC | Draw Desc | Image

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☐ 4. Document ID: US 5763101 A

L15: Entry 4 of 8 File: USPT Jun 9, 1998

US-PAT-NO: 5763101
DOCUMENT-IDENTIFIER: US 5763101 A

TITLE: Polyalcohol film and laminated film comprising the same

DATE-ISSUED: June 9, 1998

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Yoshimi; Kazuyori	Kurashiki	N/A	N/A	JPX
Michihata; Keizo	Kurashiki	N/A	N/A	JPX
Aoyama, deceased; Akimasa	late of Kurashiki	N/A	N/A	JPX

US-CL-CURRENT: 428/524; 428/910

Full | Title | Citation | Front | Review | Classification | Date | Reference | Claims | KWIC | Draw Desc | Image

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☐ 5. Document ID: US 5346950 A

L15: Entry 5 of 8 File: USPT Sep 13, 1994

wer heated to 250°–450° F., and the take-off roll 14 was cooled to obtain the desired final colorant flakes. A dough-like mixture forms on the heated rolls 10, 12 which attains a temperature of more than 212° F. thereby driving off the water and moisture and replacing the same with a hydr phobic wax matrix. The resulting paste-like mixture passes through the front two hot dispersing rolls 10, 12 to the cooling roll 14. Because of the pressure and temperature variations on the rolls, there is provided a continuous sheet which is fractured into a snowflake-like product. The small "chip-like", product or colorant can be employed as a plastic product colorant. Analysis of the resulting product shows a high pigment dispersion due to the high shear attained by the three roll mill A, as well as the excellent wetting properties of the hydroxystearate waxes. The residual moisture is normally less than 5% by weight and generally less than 3%–4%.

EXAMPLE 2

500 g of FD&C yellow #5 (containing up to 20% moisture) is admixed with 500 g of glyceryl-tris-12-hydroxystearate. The resulting mixture is treated using the aforementioned three roll mill. The resulting powdery, "nondusting" colorant or product was found to contain less than 3% moisture. By their nature, FD&C colorants normally would hygroscopically pick up about 10% moisture. However, the product produced in accordance with the invention was analyzed after one week open exposure to the atmosphere. The surprisingly low moisture absorption is contrary to any FD&C pigment colorant or product known heretofore.

EXAMPLE 3

900 g of 50% pigment scarlet presscake was admixed with 400 g of glyceryl-tris-12-hydroxystearate. The resulting mixture was treated as in Example 1, resulting in a colorant which was evaluated and found to be free of pigment agglomerates normally found in poorly dispersed organic pigment concentrates. The product was easily dispersed in a polyester thermoplastic resin by normal molding techniques.

EXAMPLE 4

800 g of 50% Anthroquinone presscake was admixed with 400 g of methyl hydroxystearate. The resulting mixture was treated as in Example 1, resulting in a granular chip dispersion, which had uniquely high color development and solubility characteristics making it suitable as a paste colorant replacement in styrene-containing polyester resin systems.

Having thus defined the invention, it is claimed:

1. A method of preparing colorant flakes comprised of hydrophilic color pigment particles embedded in a wax matrix for dry compounding with a polymeric material, said method comprising the steps of:

(a) providing particles of said color pigment dispersed in a presscake having a given moisture content, said particles having a size ranging from about 0.1 micron to about 100 microns;

(b) before dehydration of said presscake, mixing said particles with particles of a hydroxystearate wax which has a melting point temperature and is brittle below a selected lower temperature substantially below 212° F. while simultaneously heating the formed admixture to a processing temperature substantially above 212° F., by discharging said pigment particles and wax particles into the nip region f a pair of rotating rolls heated to said processing temperature, to simultaneously boil away any entrapped moisture in said pigment particles and melt the said wax to encapsulate the said pigment particles and form said particle admixture into a molten wax-pigment particle mixture;

(c) passing and compressing said mixture while at said processing temperature between said rolls in the nip thereof while they are rotated at differing angular velocities to compress and subject said mixture to high shear forces acting to effect further mixing of said pigment particles with and produce substantially uniform dispersion thereof in the said melted wax while simultaneously expelling residual moisture from said pigment particles;

(d) cooling said mixture to a temperature which renders said mixture a brittle, easily breakable solid mass by passing said mixture over and in contact with a surface having a temperature below said selected lower temperature; and,

(e) then, scraping said solid mass off said surface to break it into flakes.

2. The method as defined in claim 1 wherein said wax is selected from the class consisting of: glyceryl-tris-12-hydroxystearate, methylhydroxystearate, propylene glycol mono-hydroxystearate, glyceryl mono-hydroxystearate, ethylene glycol mono-hydroxystearate and stearyl-12-hydroxystearate.

3. A composition formed from pigment particles which are encapsulated in a hydroxystearate wax, said particles being prepared by the process of claim 1.

4. A method of preparing colorant flakes comprised of hydrophilic color pigment particles embedded in a wax matrix for dry compounding with a polymeric material, said method comprising the steps of:

(a) providing particles of said hydrophilic color pigment containing absorbed moisture and having a size ranging from about 0.1 micron to about 100 microns;

(b) providing particles of a wax having a melting point temperature substantially below 212° F. and being mechanically fracturable at approximately room temperature;

(c) before dehydrating said moisture containing pigment particles, mixing said pigment particles and wax particles and simultaneously heating the formed admixture in the nip region of a pair of rotating heated rolls at a temperature in the range of 250° F. to 450° F. and rotating at different angular velocities to simultaneously boil away any entrapped moisture in said pigment particles while at the same time melt the said particles of wax to encapsulate the said pigment particles and form said particle admixture into a molten wax-pigment particle mixture;

(d) subjecting the said heated mixture to high shear mixing and simultaneous heating by passing and compressing said mixture between the said heated rolls in the nip thereof to reduce the moisture content of said mixture below about 4% by weight and form it into a layer adhering to the faster rotating one of said heated rolls;

(e) subjecting the said heated layer mixture on said one roll to additional high shear mixing action and cooling said mixture to a solidified mass at about ro m temperature by passing and compressing said layer mixture between the said one heated roll and a cooling roll at a temperature l wer than the said

US-PAT-NO: 5346950
DOCUMENT-IDENTIFIER: US 5346950 A

TITLE: Resin composition

DATE-ISSUED: September 13, 1994

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Negi; Taichi	Kurashiki	N/A	N/A	JPX
Mochizuki; Akira	Tsukuba	N/A	N/A	JPX
Nagata; Shiro	Kurashiki	N/A	N/A	JPX
Yamasaki; Komei	Ichihara	N/A	N/A	JPX
Funaki; Keisuke	Ichihara	N/A	N/A	JPX
Sumitomo; Takashi	Ichihara	N/A	N/A	JPX

US-CL-CURRENT: 525/57; 525/241, 525/56

Full | Title | Citation | Front | Review | Classification | Date | Reference | Claims | KWIC | Draw Desc | Image

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☐ 6. Document ID: US 5089353 A

L15: Entry 6 of 8 File: USPT Feb 18, 1992

US-PAT-NO: 5089353
DOCUMENT-IDENTIFIER: US 5089353 A

TITLE: Multi-layer material having gas barrier properties

DATE-ISSUED: February 18, 1992

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Negi; Taichi	Kurashiki	N/A	N/A	JPX
Mochizuki; Akira	Tsukuba	N/A	N/A	JPX
Nagata; Shiro	Kurashiki	N/A	N/A	JPX
Yamasaki; Komei	Ichihara	N/A	N/A	JPX
Funaki; Keisuke	Ichihara	N/A	N/A	JPX
Sumitomo; Takashi	Ichihara	N/A	N/A	JPX

US-CL-CURRENT: 428/518; 264/515, 264/535, 264/DIG.33, 426/127, 426/412, 428/35.4, 428/36.7, 428/412, 428/476.3, 428/476.9, 428/483, 428/910, 525/57

Full | Title | Citation | Front | Review | Classification | Date | Reference | Claims | KWIC | Draw Desc | Image

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☐ 7. Document ID: US 4664275 A

L15: Entry 7 of 8 File: USPT May 12, 1987

fracturable temperature of said wax and rotating at an angular velocity faster than said one heated roll; and,

(f) mechanically fracturing said solidifed mass int flakes by scraping it off said cooling roll.

5. The method as defined in claim 4 wherein said wax is a hydroxystearate.

6. The method as defined in claim 4 wherein said wax is selected from the class consisting of: glyceryl-tris-12-hydroxystearate, methylhydroxystearate, propylene glycol mono-hydroxystearate, glyceryl m no-hydroxystearate, ethylene glycol mono-hydroxystearate and stearyl-12-hydroxystearate.

7. A composition formed from pigment particles which are encapsulated in a hydroxystearate wax, said particles being prepared by the process of claim 4.

* * * * *

US-PAT-NO: 4664275
DOCUMENT-IDENTIFIER: US 4664275 A

TITLE: Medical container stopper

DATE-ISSUED: May 12, 1987

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Kasai; Masaaki	Fuji	N/A	N/A	JPX
Ishikawa; Kenji	Fujinomiya	N/A	N/A	JPX

US-CL-CURRENT: 215/247

Full | Title | Citation | Front | Review | Classification | Date | Reference | Claims | KWIC | Draw Desc | Image

☐ 8. Document ID: US 4116895 A

L15: Entry 8 of 8 File: USPT Sep 26, 1978

US-PAT-NO: 4116895
DOCUMENT-IDENTIFIER: US 4116895 A

TITLE: Puncture sealant composition

DATE-ISSUED: September 26, 1978

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Kageyama; Kunio	Yokohama	N/A	N/A	JPX
Iwakura; Mituharu	Hiratsuka	N/A	N/A	JPX

US-CL-CURRENT: 524/574; 152/504, 252/72

Full | Title | Citation | Front | Review | Classification | Date | Reference | Claims | KWIC | Draw Desc | Image

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Term	Documents
((12 OR 11) AND 9).USPT,PGPB,JPAB,EPAB,DWPI,TDBD.	8

Display 10 Documents, starting with Document: 8

Display Format: CIT Change Format

US005440064A

United States Patent [19]

Agostini et al.

[11] Patent Number: 5,440,064

[45] Date of Patent: Aug. 8, 1995

[54] **PROCESS FOR THE PREPARATION OF ORGANOSILICON DISULFIDE COMPOUNDS**

[75] Inventors: **Giorgio Agostini**, Cruchten, Luxembourg; **Leon E. E. Christiaens**, Nandrin, Belgium; **Uwe E. Frank**, Ettelbruck, Luxembourg; **Thierry F. E. Materne**, Attert; **Vincent L. A. Tadino**, Liege, both of Belgium; **Friedrich Visel**, Bofferdange; **Rene J. Zimmer**, Howald, both of Luxembourg

[73] Assignee: **The Goodyear Tire & Rubber Company**, Akron, Ohio

[21] Appl. No.: **363,110**

[22] Filed: **Dec. 23, 1994**

[51] **Int. Cl.**[6] **C07F 7/08**

[52] **U.S. Cl.** **556/427**

[58] **Field of Search** 556/427

[56] **References Cited**

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4,595,740	6/1986	Panster	556/427 X

Primary Examiner—Paul F. Shaver
Attorney, Agent, or Firm—Bruce J. Hendricks

[57] **ABSTRACT**

The present invention relates to a process for the preparation of organo silicon disulfide compounds. The process involves oxidizing a mercaptoalkoxysilane in the presence of manganese dioxide.

10 Claims, No Drawings

DB Name	Query	Hit Count	Set Name
USPT,PGPB,JPAB,EPAB,DWPI,TDBD	liquid paraffin	12284	L14
USPT,PGPB,JPAB,EPAB,DWPI,TDBD	terpene resin	4194	L13
USPT,PGPB,JPAB,EPAB,DWPI,TDBD	hydrogenated styrene isoprene copolymer	46	L12
USPT,PGPB,JPAB,EPAB,DWPI,TDBD	hydrogenated styrene butadiene copolymer	162	L11
USPT,PGPB,JPAB,EPAB,DWPI,TDBD	l6 and l9	3	L10
USPT,PGPB,JPAB,EPAB,DWPI,TDBD	sealant	53604	L9
USPT,PGPB,JPAB,EPAB,DWPI,TDBD	L1 and L2 and l3 or l4 [ab]	8	L8
USPT,PGPB,JPAB,EPAB,DWPI,TDBD	L1 and L2 and l3 or l4 [ti]	0	L7
USPT,PGPB,JPAB,EPAB,DWPI,TDBD	L1 and L2 and l3 or l4	35	L6
USPT,PGPB,JPAB,EPAB,DWPI,TDBD	L1 AND L2 and l3 or l4	35	L5
USPT,PGPB,JPAB,EPAB,DWPI,TDBD	HV-300	35	L4
USPT,PGPB,JPAB,EPAB,DWPI,TDBD	HV-100	16	L3
USPT,PGPB,JPAB,EPAB,DWPI,TDBD	ESCOREZ 5320	59	L2
USPT,PGPB,JPAB,EPAB,DWPI,TDBD	KRATON G-1652	148	L1

temperature and substantially above about 212° F. to vaporize entrapped water, subjecting the mixture to high shear mixing while at or above the process temperature, cooling the mixture to a temperature below the given lower temperature to produce a solid mass, and, then, breaking the solid mass into flakes.

In accordance with another aspect of the present invention, there is provided a composition in the form of flakes, each of which includes color pigment particles each encapsulated in wax and the flakes having a water content of less than about 3%–4% by weight.

By using the above-defined invention, the resulting composition approaches an ideal colorant for use in polymeric plastic and rubber material. It is known that pigment agglomerations and aggregations are often produced in the form of pigment "presscakes". Presscakes are then dried to remove moisture and produce a relatively dry powder product. This dry product is then mixed with thermoplastic material and worked in an extruder. Thereafter, the extruded combination presscake and plastic material is chopped into pellets to form a commercial plastic stock. This is a hydrophilic type of material used as a colorant and incorporates the high moisture presscake. The presscake powder is mixed with the thermoplastic material to produce the colored plastic pellets. The extent of a dispersion of the presscake powder in the thermoplastic material is normally controlled by the amount of working done by the extruder used to extrude the plastic mass to break up the pigment agglomerates formed during the drying of the presscake. This prior system for producing a plastic colorant entraps water because of its hydrophilic nature and does not produce a finely dispersed pigment for subsequent coloring of plastic articles.

In the present invention, it is preferred to use presscake pigment before it has been dried. As is known, drying causes agglomerations due to the close natural bonding between the pigment after water is driven therefrom. Of course, dried pigment can also be used in accordance with the present invention. In the present invention, a high shear milling is employed as will be described in more detail. The special wax matrix contemplated by the present invention replaces the water binder of presscake particles with a wax binder to produce wax coated pigment particles. During the processing of the pigments, the normal water entrapped in the presscake particles is driven off by processing the mixture in an open milling container which allows rapid removal of water vapor from the pigment particles. The present invention reduces the amount of total energy consumed in the oven drying step which is normally used in drying presscake to remove its water content. In the present invention, the water content is removed simultaneously with the mixing operation. This saves a substantial amount of energy and creates a far superior end product. The direct replacement of water with a wax binder before the presscake pigment can agglomerate is a new concept in using presscake pigment. During the simultaneous driving off of water and incorporation of the special wax, which in practice is a hydroxystearate wax, pigments are protected against subsequent absorption of water. Thus, they are converted into a hydrophobic colorant having a long shelf life and which does n t create striations and other defects in subsequently formed plastic due to retained or absorbed moisture. Thus, by using the combined water extraction and wax coating process, a substantial amount of energy is saved which is both economically and environmentally advantageous.

Another advantage of the present system is that the resulting colorant having a water content of less than about 3%–4% is extremely beneficial because of the reduction in water content, the hydrophobic nature of the compound, and the extremely fine dispersion of the pigments within the colorant to produce an increased hue in the resulting plastic article. In accordance with the present invention, a three roll mill having two heated feed rolls and an adjacent cooling roll are used. The present method can be performed by a single, commercially available piece of equipment with minor modifications. The machine contemplated by the present invention to perform the method set forth above produces a one step mechanical method for producing the finished chip or flakes of colorant which have the attributes of high pigment concentration, high color hue, and low moisture affinity. The resulting chip or flakes produced by the apparatus are non-dusting in that they do not break into dust particles which would cause difficulty in meeting the OSHA regulations for industrial manufacturing plants. The present invention produces a high dispersion characteristic due to the high shear mixing obtained in the milling machine contemplated by the present invention. The concept of making color concentrate or colorant in a single step using non-agglomerated presscake has not been used in the art of producing colorants from pigments. As previously mentioned, the term "presscake" relates to pigment particles which are formed in a manner to produce the pigment which is finely divided. This pigment has a high water content of approximately 50% and agglomeration can occur if this pigment is dried preparatory to use in a process for making a colorant from the pigment. In the past, if presscake were dried, it had to be subsequently milled to again produce unagglomerated pigment for subsequent processing. This required a substantial amount of energy and processing time not necessary in the present invention which can accept and process presscake pigment without prior drying. In this manner, presscake pigment remains non-agglomerated so that there can be a direct replacement of the moisture with wax during the mixing and heating process contemplated in the present invention. The wax matrix binders contemplated by the present invention are FDA approved for contact with food. In this manner, the resulting plastic material can be formed into plastic containers for use in the food process industry which is an important feature in any colorant for plastic material. If the colorant does not have FDA approval or is formed from material not having FDA approval, this is a serious economic and practical drawback to the colorant. The present invention also contemplates the use of FDA approved "FD & C pigments". These color pigments are produced and are commercially available in presscake or other form containing generally 5–30% water. This type of FDA-approved pigment can be used in the present invention without dehydration and the resulting composition or colorant has a final water content of less than 3% by weight. Thus, the resulting product is acceptable to the FDA and has a low moisture content which is necessary to produce uniform, high-luster colors in plastic and rubber materials. The high pigment l ading obtained by the present inventi n is in excess f approximately 75–90% by weight which is apparently obtainable because of the use of the milling process which allows a thorough heating of the special

Full | Title | Citation | Front | Review | Classification | Date | Reference | Claims | KWIC | Draw Desc | Image

☐ 3. Document ID: US 4116895 A

L10: Entry 3 of 3 File: USPT Sep 26, 1978

US-PAT-NO: 4116895
DOCUMENT-IDENTIFIER: US 4116895 A

TITLE: Puncture sealant composition

DATE-ISSUED: September 26, 1978

INVENTOR-INFORMATION:

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US-CL-CURRENT: 524/574; 152/504, 252/72

Full | Title | Citation | Front | Review | Classification | Date | Reference | Claims | KWIC | Draw Desc | Image

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Term	Documents
(6 AND 9).USPT,PGPB,JPAB,EPAB,DWPI,TDBD.	3

Display 10 Documents, starting with Document: 3

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United States Patent [19]

Pollard

[11] **4,173,492**

[45] **N v. 6, 1979**

[54] **METHOD OF PREPARING COATED PIGMENT PARTICLES AND THE PRODUCT PRODUCED THEREBY**

[76] Inventor: **Edward T. Pollard**, 78 Jefferson, Huron, Ohio 44839

[21] Appl. No.: **888,946**

[22] Filed: **Mar. 22, 1978**

Related U.S. Application Data

[63] Continuation of Ser. No. 668,724, Mar. 19, 1976, abandoned.

[51] **Int. Cl.**[2] **C09C 3/08**

[52] **U.S. Cl.** ..... **106/308 F**; 106/308 Q; 252/316

[58] **Field of Search** ..... 252/316; 106/308 F, 106/308 Q; 428/403; 427/221, 374 C; 366/71, 144

[56] **References Cited**

U.S. PATENT DOCUMENTS

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The Condensed Chemical Dictionary (van Nostrand Reinhold Company, New York), 1971, pp. 176-177, "castorwax".

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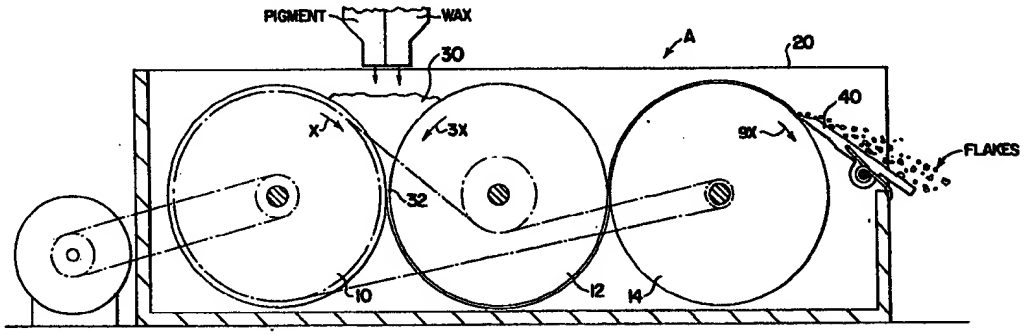
[57] **ABSTRACT**

A method of preparing coated pigment particles for dry compounding with a polymeric material such as plastic or rubber, which method comprises the steps of providing particles of a hydrophilic color pigment, mixing the pigment particles with a wax having a given melting point temperature and a brittle condition below a given lower temperature, heating the mixture above a process temperature substantially above the melting temperature and substantially above the boiling point of water to boil away entrapped water, subjecting the mixture to high shear mixing while at the process temperature, cooling the mixture to a temperature below the given lower temperature to produce a solid mass, and, then, breaking the solid mass into flakes.

7 Claims, 2 Drawing Figures



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☐ 1. Document ID: US 5578142 A

L10: Entry 1 of 3 File: USPT Nov 26, 1996

US-PAT-NO: 5578142
DOCUMENT-IDENTIFIER: US 5578142 A

TITLE: Solar-cell module and process for producing the same

DATE-ISSUED: November 26, 1996

INVENTOR-INFORMATION:

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US-CL-CURRENT: 136/251; 156/303.1, 156/331.4, 257/433, 438/118, 438/64

Full | Title | Citation | Front | Review | Classification | Date | Reference | Claims | KWIC | Draw Desc | Image

☐ 2. Document ID: US 4455146 A

L10: Entry 2 of 3 File: USPT Jun 19, 1984

US-PAT-NO: 4455146
DOCUMENT-IDENTIFIER: US 4455146 A

TITLE: Novel plasters

DATE-ISSUED: June 19, 1984

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US-CL-CURRENT: 424/448; 156/231, 156/246, 156/247, 156/249, 156/289, 156/334, 424/447, 424/449, 428/349, 428/352, 428/354, 428/355BL, 428/355CP, 602/903, 604/290, 604/304

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